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## Synthesis and Characterization of Some Metals Complexes with New Acidicazo Ligand 4-[(2-Amino-4-Phenylazo)-Methyl]-Cyclohexane Carboxylic Acid

Rasha Khider Hussain Al-Daffay<sup>2</sup>, Abbas Ali Salih Al-Hamdani<sup>1</sup>

<sup>1</sup>Department of Chemistry, College of Science for Women, University of Baghdad, Baghdad, Iraq

<sup>2</sup>Department of Chemistry, College of Science, University of Baghdad, Baghdad, Iraq

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### Abstract

4-[(2-Amino-4-phenylazo)-methyl]-cyclo hexane carboxylic acid, a new Azo (LH2) ligand, was synthesized by reaction of the diazonium salt of trans-4-(amino-methyl) cyclo hexane carboxylic acid with 3-amino phenol. The azo ligand was characterized by micro elemental analysis (C.H.N.O.) and TGA as well as spectroscopic techniques (UV-Vis, FTIR, <sup>1</sup>H-NMR, and LC-Mass). Atomic absorption, elemental analysis, infrared, LC-Mass, TGA, and UV-Vis spectral methods, as well as conductivity and magnetic susceptibility, were used to characterize the complexes of Ni (II), Pd (II), and Pt (IV). The following general formula has been given for the produced compounds based on the results obtained, which are as following: the Ni (II) complex has a 1:1 metal-ligand ratio, but the Pd (II) and Pt (IV) complexes have a 1:2 metal-ligand ratio.

**Keywords:** Azo ligand acid, Mass spectroscopy, Metal complexes, Tranexamic acid

تحضير وتشخيص بعض معقدات الفلزات مع ليكاند آزو حامضي جديدة 4-((2-امينو-4-فينيل آزو)-  
مثيل)-سايكلوهكسان حامض كربوكسيل

رشا خضر حسين الدفاعي<sup>2</sup>، عباس علي صالح الحمداني<sup>1</sup>

<sup>1</sup>قسم الكيمياء، كلية العلوم للبنات، جامعة بغداد، بغداد، العراق

<sup>2</sup>قسم الكيمياء، كلية العلوم، جامعة بغداد، بغداد، العراق

### الخلاصة

حضرت معقدات جديدة للنكل والبلاديوم والبلاتين باستخدام ليكاند الأزو الجديد 4-((2-امينو-4-فينيل آزو)-مثيل)-سايكلوهكسان حامض كربوكسيل تم تشخيص المركبات المحضرة باطياف (الأشعة تحت الحمراء الأشعة فوق البنفسجية - المرئية والكتلة والرنين النووي المغناطيسي للبروتوني) إضافة إلى التحليل الدقيق للعناصر ومنحنى التحلل الحراري الوزني مع قياس التوصيلية المولارية وتحديد نسبة الفلز، أعطت نتائج التحليل أن الليكاند يسلك ثنائي السن مع النكل والبلاديوم بينما ثلاثي السن مع البلاتين وقد أعطى معقد النكل رباعي السطوح بينما معقد البلاديوم مربع مستوي ومعقد البلاتين ثماني السطوح وبينت دراسة التحلل الحراري استقرار المركبات حرارياً ووجود جزيئات ماء داخل مجال التناسق.

\*Email: [rasha.khedr@sc.uobaghdad.edu.iq](mailto:rasha.khedr@sc.uobaghdad.edu.iq)

## 1. Introduction

Numerous industries utilize aromatic azo compounds extensively. For instance, the azo compounds are employed as organic dyes, markers, radical reaction catalysts, therapeutics, and drug delivery systems [1]. They are also important in different area, azo compounds (R-N=N-R) are important chemical compounds used in a wide range of products, such as organic reagents, paints, food coloring, and color filters for LCD displays [2]. Among the biological functions of azo compounds, which also include antibacterial, antifungal, pesticidal, antiviral, and anti-inflammatory characteristics, are antitumor, antioxidant, drug delivery compounds, and drug polymers [3-5]. A lot of interest has been paid to azo compounds metal complexes because of their applications in dyes, pigments, functional materials, and optical computing [6]. Azo compounds have various efficient groups that may form coordination complexes with a variety of metal ions, making them helpful in a wide range of chemical analytical fields. Preparing these substances and examining their characteristics and efficacy, notably the differences, have attracted the attention of several researchers.[7]

Trans-4-(aminomethyl) cyclohexane carboxylic acid, with the formula  $C_8H_{15}NO_2$ , is the chemical name for tranexamic acid. It is the closed cyclic analog of lysine [8–10]. It has been routinely used to prevent excessive blood loss during both normal surgery and trauma because it has strong anti-fibrinolytic characteristics [11–13]. The main objective of the current study is the 4-[(2-Amino-4-phenylazo)-methyl] cyclohexane carboxylic acid and its Ni (II), Pd (II), and Pt (IV) complexes were synthesized and characterized using spectrum methods, micro elemental analysis, TGA, and for complexes, using atomic absorption, elemental analysis, as well as conductivity and magnetic quantifications.

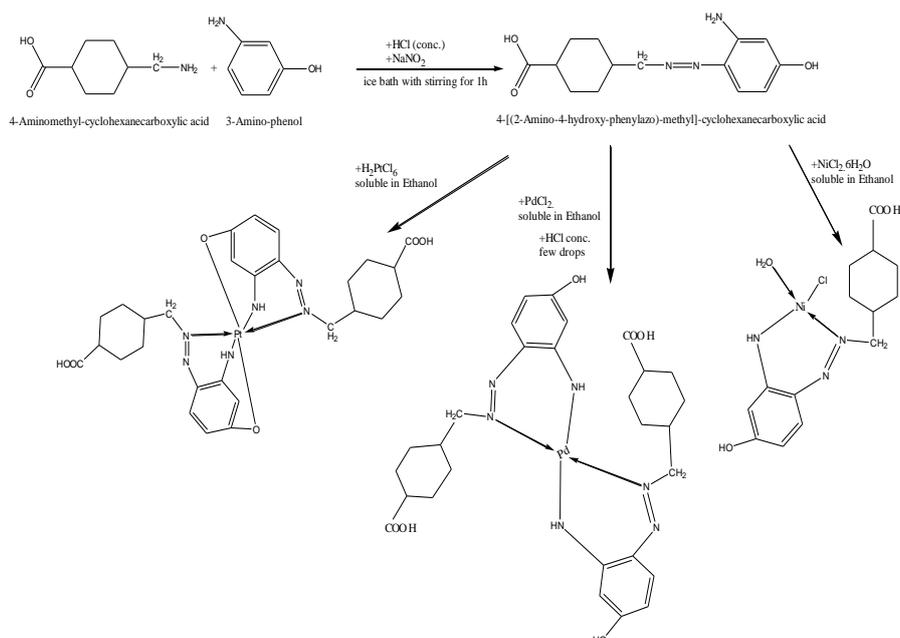
## 2. Experimental

### 2.1. Materials and instrumentation

All chemicals and reagents were purchased from commercial sources (Sigma-Aldrich, Merck, and others). The Euro vector model EA/3000, single-V.3.O-single, was used to conduct elemental analyses (C, H, and N). Metal ions were estimated as metal oxides using a gravimetric method. The complexes' molar conductance was measured using a Conductometer WTW at 25°C at a concentration of  $1 \times 10^{-3}$  M. Dimethylformamide was used to dissolve all of the complexes (DMF). A Mass spectrometry (MS) type QP50A: DI Analysis Shimadzu QP-2010-Plus (E170Ev) was used for conducting all the mass measurements of the target molecules. The UV-Vis spectrophotometer type 1800 Shimadzu was used to obtain the wavelengths of the molecules. A Bruker 300 MHz was used to record the proton nuclear magnetic resonance ( $^1H$ -NMR) spectra for ligand in DMSO- $d_6$ . The IR Prestige-21 was used to investigate the Fourier transform infrared (FTIR) spectra, and the Perkin-Elmer Pyris Diamond TGA was used to conduct thermogravimetric studies.

### 2.2. Synthesis of azo dye ligand: 4-[(2-Amino-4-phenylazo)-methyl]-cyclohexane carboxylic acid

Tranexamic acid (0.1577 g, 1.0 mmol/L) was melted in a solution of 5 mL ethanol and 3 mL concentrated HCl before being diazotized at 5 °C with a 10 percent  $NaNO_2$  solution. For 3-aminophenol (0.109 gm, 1.0 mmol/L), a diazotized solution was added to a cooled ethanolic solution while being stirred at room temperature. After direct merger, a dark colored mix and azo ligand precipitation were seen. This deposit underwent filtering, several ounces of washing, and drying for a (1:1) ( $C_2H_5OH$ :  $H_2O$ ) combination. The solution is shown in Scheme (1) [14].



**Scheme 1.** Synthesis of azo dye ligand and metal complexes

### 2.3. General method for the preparation of metallic ions complexes

Metal chlorides were used to generate the metallic ions complexes for Ni (II), Pd (II), and Pt (IV). To a stoichiometric mixture of (1.0 mmol/L) of [1:1] M: L for Ni (II) chloride salt and (0.5m.mol) of [1:2] M: L for Pd (II) and Pt, a stoichiometric amount of 0.331g (1m.mol) from azo ligand, dissolved in 10 ml 100% ethanol, was progressively added in drops with shaking (IV) After being heated for two hours at (50–70) °C, the mixture was cooled in an ice bath till precipitation formed, and then left overnight. The solid complexes were separated and rinsed with distilled water and a tiny quantity of hot ethanol to get rid of any residual constituents. Lastly, the complexes were dried using vacuum desiccators. The analytical and physical properties of the ligand and its metal complexes are summarized in Table 1.

**Table 1-** Physical properties & analytical data of ligand & their complexes

Comp.	Chemical Formula M. wt	Color	m.p	Elemental microanalysis%					
				C Found	H Found	N Found	O Found	M Found	Cl Found
				C Calc	H Calc.	N Calc.	O Calc.	M Calc.	Cl Calc.
LH <sub>2</sub>	C <sub>14</sub> H <sub>19</sub> N <sub>3</sub> O <sub>3</sub> 276	Brown	280- 282	62.11	5.02	17.05	16.03	-	-
				60.6	6.85	15.16	17.3	-	-
[NiLH (H <sub>2</sub> O) Cl]	C <sub>14</sub> H <sub>20</sub> ClN <sub>3</sub> NiO <sub>4</sub> 388	Brown	>300	42.45	3.77	12.71	18.21	16.88	9.54
				43.29	5.15	10.83	16.49	15.13	9.15
[Pd (LH) <sub>2</sub> ]	C <sub>28</sub> H <sub>36</sub> N <sub>6</sub> PdO <sub>6</sub> 659	Deep Brown	>300	52.11	4.10	14.42	13.10	18.01	-
				50.99	5.46	12.74	14.56	16.15	-
[Pt (LH) <sub>2</sub> ]	C <sub>28</sub> H <sub>34</sub> N <sub>6</sub> PtO <sub>6</sub> 745	Brown	>300	44.74	4.01	12.74	13.74	25.08	-
				45.1	4.563	4.56	11.27	26.19	-

## 3. Result and discussion

### 3.1. Physical and chemical properties of azo dye ligand

The amorphous appearance of the azo dye ligand (LH<sub>2</sub>), which gives the appearance of a fine brown powder, makes it stand out. This synthetic ligand is soluble in both water and DMSO,

although it dissolves in ethanol much more easily. The azo ligand and metallic ions complexes that were created were stable in the presence of air.

### 3.2. $^1\text{H-NMR}$ spectra

The ligand  $\text{LH}_2$ 's  $^1\text{H-NMR}$  spectra in  $\text{DMSO-d}_6$  are displayed in Figure 1. The spectra showed a peak at  $\delta$  (1.38) ppm, which was attributed to chemical reactions involving  $-\text{N}=\text{N}-\text{CH}_2$ . The peaks at  $\delta$  (1.9) ppm were attributed to the chemical shift of  $(\text{CH}_2-\text{CH}_2)$  protons on Tranexamic acid. The  $\text{CH}_2-\text{COO}$  proton in the cyclohexane ring was linked to several signals at  $\delta$  (2.69) ppm for the ligand. The  $\text{NH}_2$  group is visible as a singlet at 4.61 ppm. The greatest signal, at  $\delta$  9.16 ppm, was attributed to the proton (OH) phenolic ring. The numerous peaks at  $\delta$ , (6.82-7.56) ppm are ascribed to the aromatic protons of benzene groups. The singlet signal at  $\delta$  (11.49) ppm is caused by the proton (OH) of the carboxyl group  $\text{COOH}$  [15].

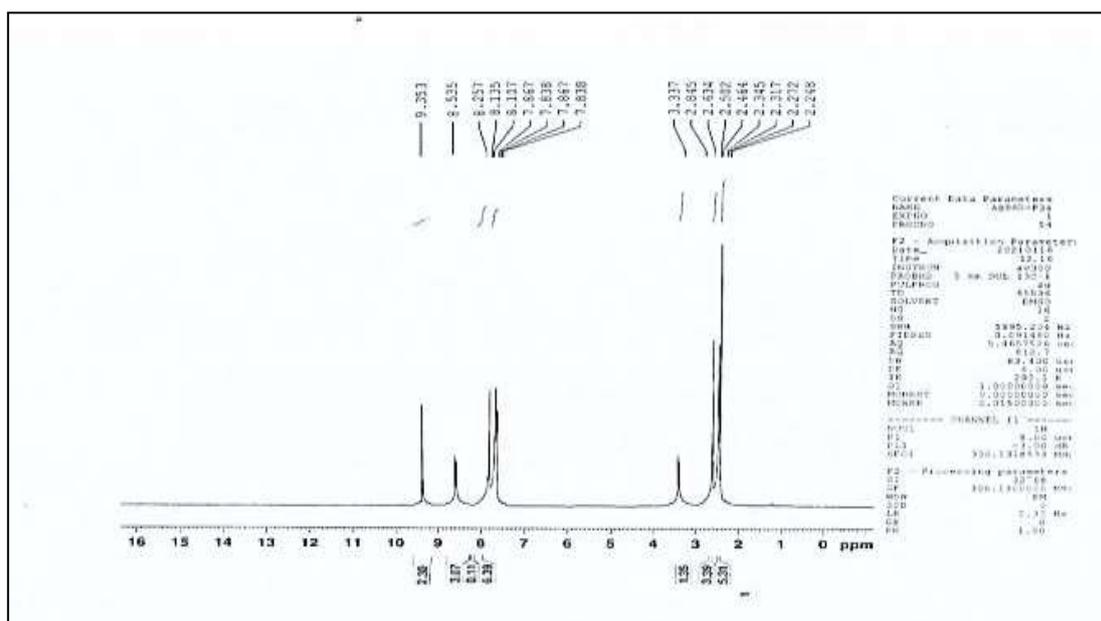


Figure 1- $^1\text{H-NMR}$  spectrum of ligand

### 3.3. Electronic spectra measurements

The UV-Vis spectra of the ligand  $\text{LH}_2$  and its complexes are displayed in Table 2 and Figure 2. A peak with a high intensity band produced with absorption maxima at (302 nm,  $33112.5 \text{ cm}^{-1}$ ) attributable to the  $n \rightarrow \pi^*$  transition, a peak with a high intensity band formed with absorption maxima at (302 nm,  $33112.5 \text{ cm}^{-1}$ ). The  $[\text{Ni}(\text{L})(\text{H}_2\text{O})\text{Cl}]$  complex's electronic spectra showed three absorption peaks in Figure 3. The peak at 372 nm is assigned to the ligand, while the peaks from the (C.T) complexes and one peak at 670 are due to the (d-d) electronic transitions types  $^3\text{T}_1 \rightarrow ^3\text{T}_{1(\text{P})}$ . Furthermore, the magnetic moment of the Ni (II) ( $d^8$ ) complexes is found to be 3.9. The tetrahedral geometry is consistent with all of the aforementioned data [10].

The peak at 313 nm and the peak at 648 nm with shoulders at range (375-494) nm, respectively, were assigned to the electronic spectra of the diamagnetic Pd (II) complex and suggest a square planar structure in Figure 4 ( $^1\text{A}_{1\text{g}} \rightarrow ^1\text{A}_{2\text{g}}$  and  $^1\text{A}_{1\text{g}} \rightarrow ^1\text{B}_{1\text{g}}$  transitions, respectively). While the electronic spectra of the Pt (IV) complex revealed three peaks at 367 nm corresponding to the ligand field and peaks at 537 and 654 nm corresponding to  $^1\text{A}_{1\text{g}} \rightarrow ^1\text{T}_{2\text{g}}$  and  $^1\text{A}_{1\text{g}} \rightarrow ^1\text{T}_{1\text{g}}$ , respectively indicating an octahedral geometry and the Pt (IV) complex was diamagnetic [16, 17].

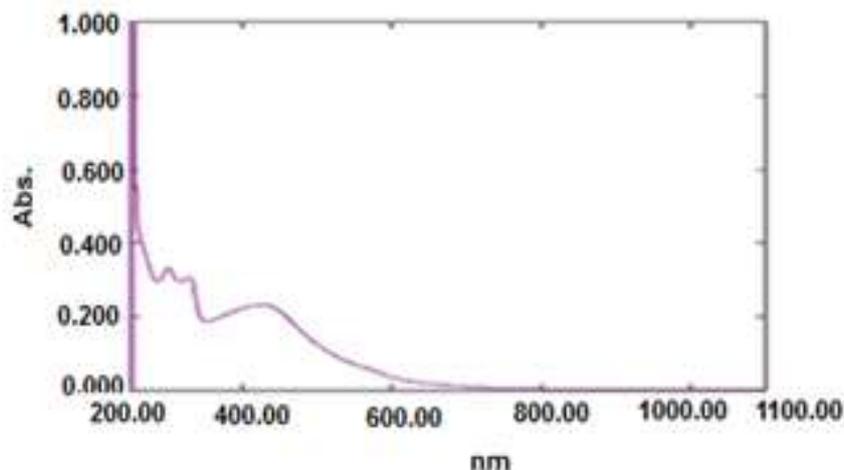


Figure 2-Electronic spectra of azo ligand

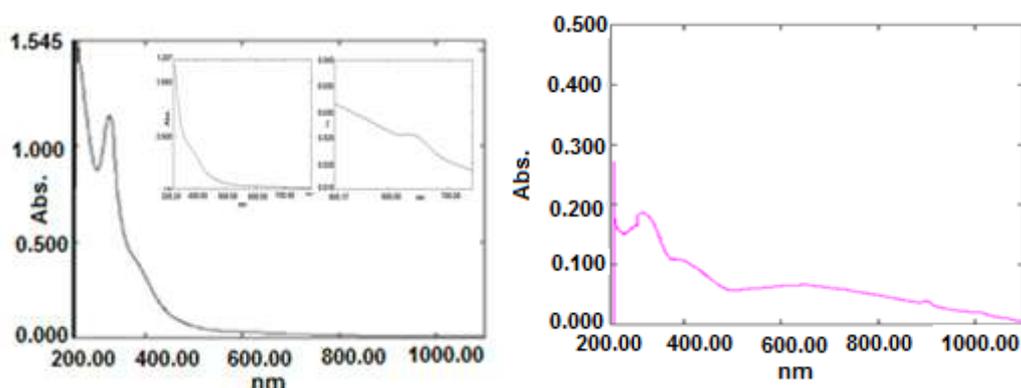


Figure 3-Electronic spectra of Ni (II) complex Figure 4-Electronic spectra of Pd (II) complex

Table 2- Electronic spectral data of the metal complexes with LH<sub>2</sub> ligand and molar conductivity in DMSO ( $1 \times 10^{-3}$  M)

Complexes Geometry	$\lambda_{\max}$ (nm)	$\nu_{\text{cm}^{-1}}$	ABS	$\epsilon_{\max} L$ $\text{mol}^{-1}\text{cm}^{-1}$	Assignment	$\Lambda_m \text{cm}^2$ $\Omega^{-1}\text{mol}^{-1}$
LH <sub>2</sub>	302	33113	0.248	248	$\pi \rightarrow \pi^*$	-
	330	30303	0.235	235	$n \rightarrow \pi^*$	
	426	23474	0.204	204	$n \rightarrow \pi^*$	
[Ni (LH) (H <sub>2</sub> O) Cl] Tetrahedral	327	30581	1.17	1170	$\pi \rightarrow \pi^*$	12
	(362-445)	27624-22472	0.477	477	C.T.	
	670	14925	0.26	260	${}^3T_{1(F)} \rightarrow {}^3T_{2(P)}$	
[Pd (LH) <sub>2</sub> ] Square planar	318	31447	0.188	188	$\pi \rightarrow \pi^*$	16
	(375-494)	266667-20243	0.109	109	${}^1A_{1g} \rightarrow {}^1A_{2g}$	
	648	15432	0.067	67	${}^1A_{1g} \rightarrow {}^1B_{1g}$	
[Pt(L) <sub>2</sub> ] Octahedral	258	38760	0.338	338	$\pi \rightarrow \pi^*$	17
	367	27248	0.077	77	$\pi \rightarrow \pi^*$	
	537	18622	0.047	47	${}^1A_{1g} \rightarrow {}^1T_{2g}$	
	654	15291	0.035	35	${}^1A_{1g} \rightarrow {}^1T_{1g}$	

### 3.4. Liquid chromatography–mass spectrometry (LC–MS) measurements

To obtain the mass spectra of the new ligand and metal complexes, electron impact of fragmentation was employed. In general, substantial fragments associated to breakdown products as well as the free azo ligand and its complexes were recovered using high-

resolution MS [18]. The electron impact mass spectrum of the ligand LH<sub>2</sub> is seen in Figure 5. The predicted molecular weight of this ligand is 277 g/mol. A signal at 276 m/z in the spectra was assigned to [M]<sup>+</sup> and related to the new azo moiety C<sub>14</sub>H<sub>19</sub>N<sub>3</sub>O<sub>3</sub>. Other fragments may be responsible for the characteristic peaks at 137, 84, and 55 m/z. Their brilliance denotes the stability of the parts. The mass spectrum of the Ni (II) complex is shown in Figure 6. The complex moiety C<sub>14</sub>H<sub>20</sub>ClN<sub>3</sub>NiO<sub>4</sub> in the spectrum showed a peak at 388 m/z, which matched the peak of the complex moiety. The 261 and 127 m/z peaks at various frequencies might be attributable to other parts. The mass spectrum of the Pd (II) complex is shown in Figure 7. A peak at 659 m/z in the spectra allowed for the identification of the chemical moiety C<sub>28</sub>H<sub>36</sub>N<sub>6</sub>O<sub>6</sub>Pd. Other components may be responsible for the characteristic peaks at 256, 150, 123, and 119 m/z. The Pt (IV) complex's electron impact mass spectrum is shown in Figure 8. A signal at 745 m/z in the spectra helped to identify the complex moiety C<sub>30</sub>H<sub>28</sub>N<sub>2</sub>O<sub>6</sub>PtCl<sub>2</sub>. Other distinctive peaks at 359, 150, 123, and 106 m/z might be attributed to other pieces. In Schemes 2–5, suggested fragmentation routes and structural assignments of pieces are given.

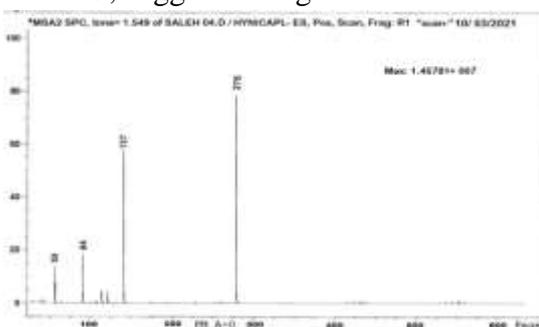


Figure 5- (LC-MS) spectrum of ligand

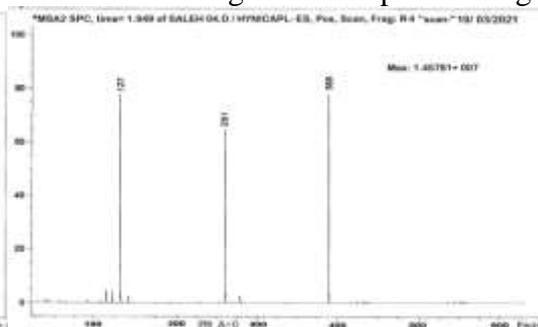


Figure 6 -(LC-MS) spectrum of Nicomplex

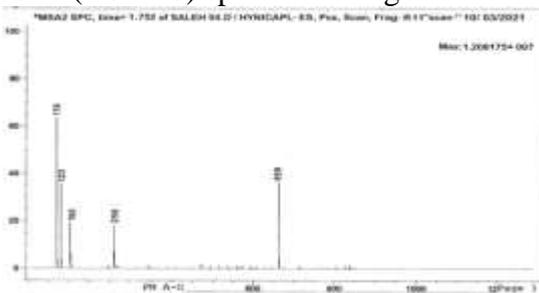


Figure 7-(LC-MS) spectrum of Pd complex

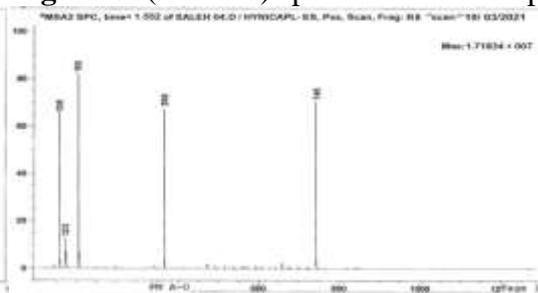
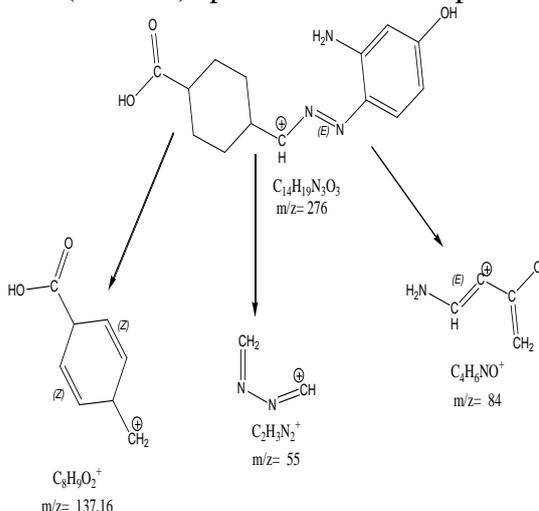
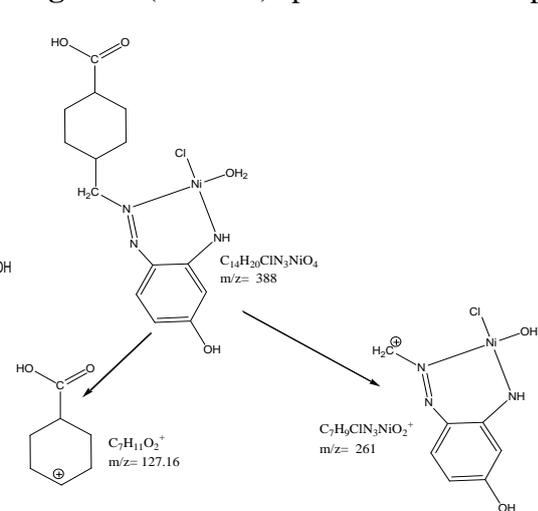


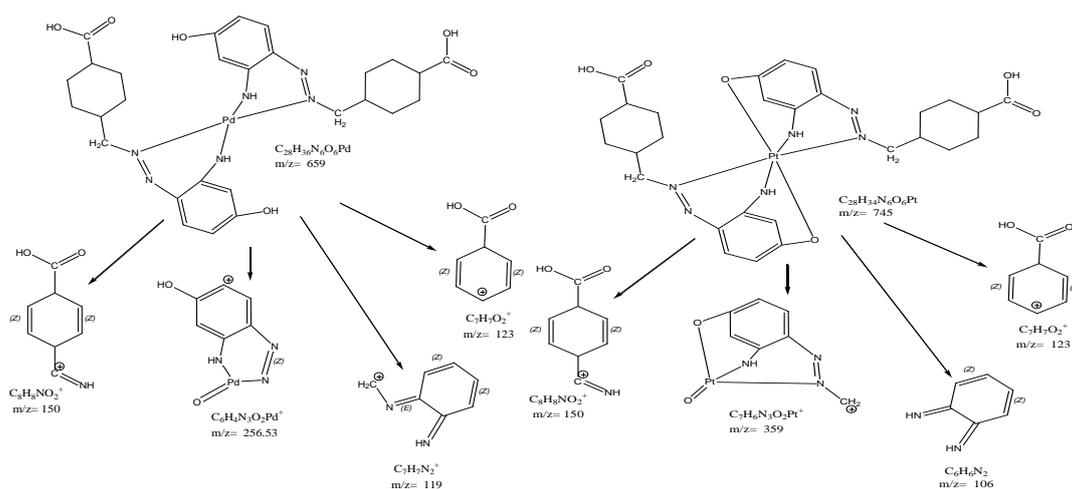
Figure 8-(LC-MS) spectrum of Pt complex



Scheme -2 Fragmentation pattern of ligand



Scheme -3 Fragmentation pattern of Ni complex



**Scheme -4** Fragmentation pattern of pd complex    **Scheme -5** Fragmentation pattern of pt complex

### 3.5. Infrared spectra measurements

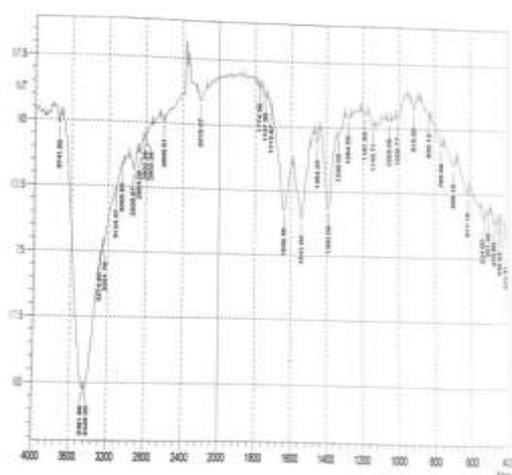
FTIR data was utilized to identify the functional groups in molecules, particularly organic compounds, when coordination occurs via the modification of the functional group frequencies (that have the donor atom) [19]. In some instances, when coordination occurs through the fluctuation of the functional group frequencies, it can really provide suggestions for complexes formation (that have the donating atom). The data has been collated and is presented in Table 3 along with the spectra of the azo ligands and their metal chelates complexes with Ni (II), Pd (II), and Pt (IV). The ligand had bands at 3429 and 3275  $\text{cm}^{-1}$  that were attributed to the (NH<sub>2</sub>) stretching mode, but these bands were attenuated to a lower frequency in the spectra of all synthesized compounds, indicating coordination with a metal ion [20]. The (N=N) stretching vibration was attributed to the band seen at 1454  $\text{cm}^{-1}$  [21] in the unbound azo ligand (LH<sub>2</sub>). This band was discovered in the compounds' spectra around 1454-1456  $\text{cm}^{-1}$ . The engagement of the azo group in chelation was verified by a change in the azo group of the azo ligand [21,22]. Additionally, earlier research has demonstrated that the azo-dye nitrogen is always more likely to promote complexity in the presence of transition metals [23]. It was challenging to prove that this group was engaged in chelate formation because of the coordinated water molecules present in the Ni (II) complex. The presence of OH bands in the IR spectra of the Ni (II) complex in the 3442  $\text{cm}^{-1}$  was attributed to the presence of coordinated water molecules in the coordination sphere. Additionally, it was demonstrated that stretching vibrations between 833 and 696  $\text{cm}^{-1}$  related to  $\nu$  (M-OH<sub>2</sub>). The fact that water molecules are participating in the coordinating process is strongly supported by this [24].

For the unbound ligand, the IR spectra revealed a wide stretching vibration band at 3462  $\text{cm}^{-1}$ , which might correlate to the OH of the phenolic group [25]. The band at 1284  $\text{cm}^{-1}$  was assigned to the  $\nu$ (C-O) stretching vibration of the phenolic group of the free azo-dye ligand due to the coordination with the Pt (IV) ion [26], and the band at 1284  $\text{cm}^{-1}$  was attributed to the  $\nu$ (C-O) stretching vibration of the phenolic group of the free azo-dye ligand. This band shifted in the Pt complex, showing that the deprotonated phenolic OH group is involved in coordination [27]. When comparing the spectra of all complexes to the free ligand, new bands emerged solely in the produced complexes, indicating that they had been successfully synthesized. In the Ni (II) complex, three bands at (510,466), 420, and 383  $\text{cm}^{-1}$  were attributed to  $\nu$  (M-N),  $\nu$  (M-O), and  $\nu$  (M-Cl), respectively. In the Pt (IV) complex, two bands at (568,443) and 410-416  $\text{cm}^{-1}$  were attributed to  $\nu$  (M-N) and  $\nu$  (M-O), respectively [10, 21, 28] and  $\nu$  (M-N) in the Pd (II) complex was given two bands at 487 and 466  $\text{cm}^{-1}$ . The

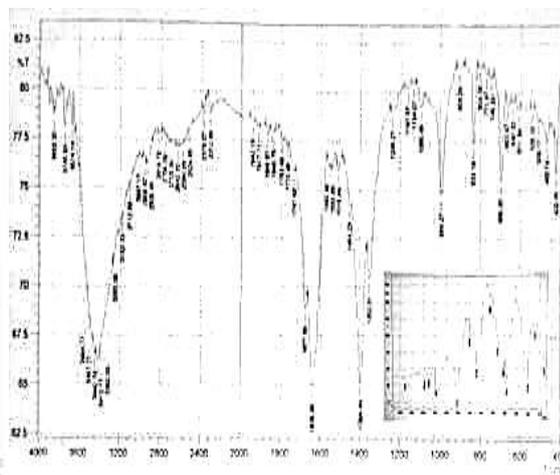
ligand and its metal complexes' IR spectra are displayed in Figures 9 and 10. The azo-dye ligand then coupled to metal ions through three sites, including the nitrogen site of the azo group, the main amine, and the oxygen site via deprotonation of the amine and phenolic groups, as shown by the IR spectra of all the compounds generated [16,21,28]. As a result, in the Pt complex, the ligand behaved as an N, N, orientate ligand, whereas in the Ni and Pd complexes, it behaved as an N, N bidentate ligand. The produced complexes spectra reveal a strong absorption band at  $1666\text{ cm}^{-1}$  owing to  $\nu(\text{C}=\text{O})$  of carboxylic group, which is detected with a little change in shape and moved to higher frequencies  $1676\text{-}1670\text{ cm}^{-1}$ . These discrepancies point to hydrogen bonding between the carboxylic group's  $\text{C}=\text{O}$  and the orthogonal  $\text{OH}$  group. The azo group was shifted toward lower frequencies in their complex's spectra, and the primary amine group appeared within the  $3440\text{ cm}^{-1}$  region, as well as the disappearance of the resorcinol hydroxyl group. These findings can be explained by the participation of  $\text{NH}$  & azo- nitrogen in coordination with metallic ions, as seen in complexes spectra [20-24,29,30].

**Table 3-** The IR spectra bands ( $\text{cm}^{-1}$ ) of the free ligand and its complexes

Comp.	$\nu\text{OH}$ phenolic	$\nu\text{OH}$ carboxylic	$\nu(\text{NH}_2)$	$\nu(\text{NH})$	$\nu\text{CO}$ carboxylic	$\nu(\text{N}=\text{N})$	$\nu(\text{H}_2\text{O})$ Coord.	Other bands
$\text{LH}_2$	3462	3252	3429 3275	-	1639	1454		-
$[\text{NiLH}(\text{H}_2\text{O})\text{Cl}]$	3467	3265	-	3414	1693	1454	3442 833 696	$\nu\text{ M-}(509,466)$ $\nu\text{ M-O}(420)$ $\nu\text{ M-Cl}(383)$
$[\text{Pd}(\text{LH}_2)]$	3477	3201	-	3407	1635	1458	-	$\nu\text{ M-}(487,466)$
$[\text{Pt}(\text{LH}_2)]$	3485	3218	-	3421	1369	1456	-	$\nu\text{ M-}(468,443)$ $\nu\text{ M-O}(416)$



**Figure -9** FT-IR spectrum of ligand  $\text{LH}_2$



**Figure -10** FT-IR spectrum of Ni-complex

### 3.6. Thermal measurements

Figures (11-13) show the TG and DTG findings of thermal decomposition of the ligand  $\text{LH}_2$  and their metal complexes. Table 4 contains information on the thermal degradation process. The prepared complexes displayed a decomposition in the thermo-gravimetric decomposition curve, with the ligand exhibiting little thermal stability at  $50^\circ\text{C}$ , comparable to the little stability complexes in the range of  $30^\circ\text{C}$  for Pd and  $40^\circ\text{C}$  for Ni complexes, indicating the presence of water molecules in the Ni complex, whether water hydrate or aqua. According to the data in Table 4, the ligand decomposes in three degrees while leaving an intact fraction,

and Ni decomposes in three ranges with an intact residue. The Pd complex decomposes in two phases with an intact residue, but only in two stages. This matches both the calculated values and the suggested formula [31-37].

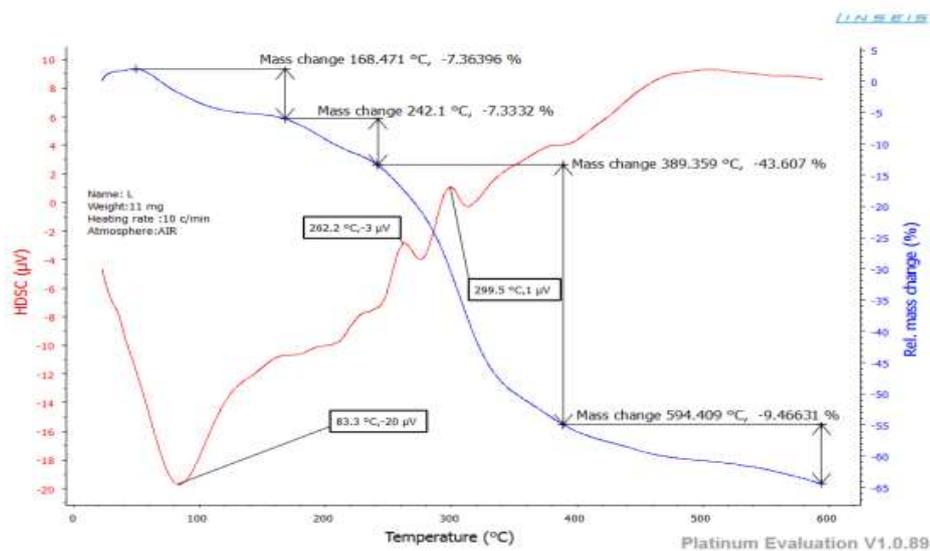


Figure 11-Thermogravimetry of Ligand

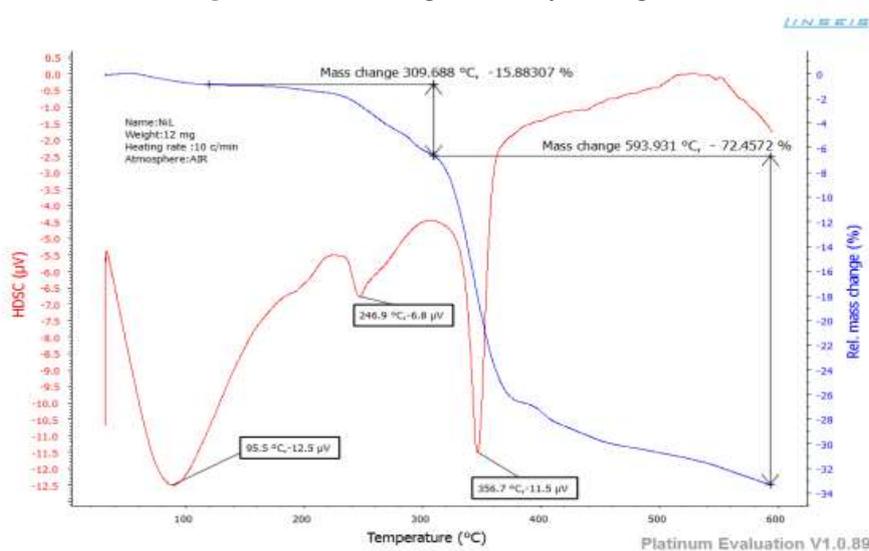


Figure 12-Thermogravimetry of Ni-complex

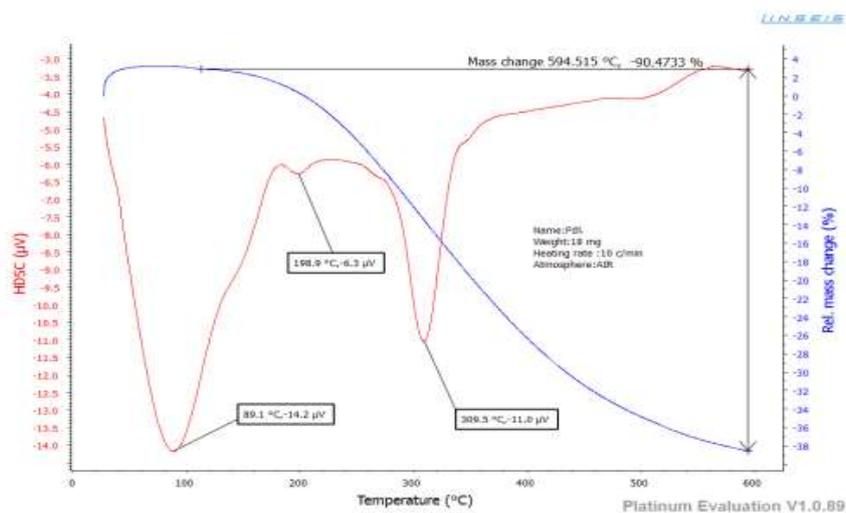


Figure 13-Thermogravimetry of Pd-complex

**Table 4-** Thermal decomposition of Ligand and its complexes

Compound	Thermogravimetric range °C	DSC max °C	% Estimated (calculated)		Assignment
			Mass loss	Total mass loss	
(L <sub>1</sub> )	50-170 170-241 241-389 389-600	83.3(Endo) 262.2(Exo) 299.5(Exo) 390(Endo) 500(Exo)	7.3(6.5) 7.33(5.8) 43.6(45.8) 9.5(9.7) 32.13 (32.22)	92.53 (93.52)	-H <sub>2</sub> O -NH <sub>2</sub> -C <sub>7</sub> H <sub>11</sub> O <sub>2</sub> -CHN
[Ni L H (H <sub>2</sub> O) Cl]	40-102 102-310 310-595	95.5(Endo) 246.9(Endo) 356.7(Endo) 522(Exo)	2.5(2.3) 15.88(16.46) 72.46(71.78) 9.16(9.66)	90.84 (90.54)	-0.5H <sub>2</sub> O -C <sub>3</sub> H <sub>8</sub> O -C <sub>11</sub> H <sub>11</sub> ClN <sub>3</sub> O <sub>2</sub> (0.5Ni) -0.5NiO
[Pd (LH) <sub>2</sub> ]	30-120 120-595	89.1(Endo) 198.9(Endo) 309.5(Endo) 560(Exo)	1.22(1.37) 90.47(89.4) 8.31(9.26)	91.69 (90.77)	-0.5H <sub>2</sub> O -C <sub>28</sub> H <sub>33</sub> N <sub>6</sub> O <sub>5</sub> (0.5Pd) -0.5PdO

### Conclusion

A completely new acid dye that has never been produced before serves as the ligand. The ligand and its complexes were identified using <sup>1</sup>H-NMR, IR, UV-Vis, TGA, and mass spectral methods. It was discovered that there was a good correlation between the estimated values and the elemental analysis' findings. The IR data revealed that the ligand had a tridentate nature. Multinuclear NMR data show that -N, N O moiety is the mechanism of complexity. Because complex molecules are more stable, less machinery is needed to accelerate the process.

### References

- [1] G. G. Mohamd, W. H. Mahmoud and A. M. Refaat, "Nano-Azo Ligand and Its Superhydrophobic Complexes: Synthesis, Characterization, DFT, Contact Angle, Molecular Docking, and Antimicrobial Studies," *Journal of Chemistry*, vol. 2020, pp. 1-9, 2020.
- [2] S. M. Mahdi and A. K. Ismail, "Preparation and Identification of new azo-schiff base ligand (NASAR) and its divalent transition metal Complexes," *Journal of Pharmaceutical Sciences and Research*, vol. 10, no. 9, pp. 2175-2178, 2018.
- [3] A. A. S. Al-Hamdani and S. A. Shaker, "Synthesis, Characterization, Structural Studies and Biological Activity of a New Schiff Base- Azo Ligand and its Complexation with Selected Metal Ions," *Oriental Journal of Chemistry*, vol. 27, no. 3, pp. 835-845, 2011.
- [4] S. Slassi, A. Fix-Tailler, G. Larcher, A. Amine and A. El-Ghayoury, " Imidazole and Azo-Based Schiff Bases Ligands as Highly Active Antifungal and Antioxidant Components," *Heteroatom Chemistry*, vol. 2019, pp. 1-8, 2019.
- [5] H. M. Alabidi, M. A. Alabidi and N. F. Makki, " Synthesis And Spectroscopic Studying of New Azo Ligand From 2-Naphthol Derivative and Its Complexes with Some Transition Metal Ions," *AL-Qadisiyah Journal of pure Science*, vol. 23, no. 1, pp. 186-195, 2018.
- [6] M. Y. Dhamra, " Spectrophotometric Determination of Tranexamic Acid by Azo-Dye Formation- Application to Pharmaceutical Preparations," *Journal of Education and Science*, vol. 24, no. 3, pp. 21-33, 2011.
- [7] N. Sher, N. Fatima, Sh. Perveen, F. A. Siddiqui and A. W. Sial, " Pregabalin and Tranexamic Acid Evaluation by Two Simple and Sensitive Spectrophotometric Methods," *International Journal of Analytical Chemistry*, vol. 2015, no. 24141, pp. 1- 7, 2015.
- [8] Z. Li, L. Fang, J. Wang, L. Dong, Y. Guo and Y. Xie, " An Improved and Practical Synthesis of Tranexamic Acid," *Organic Process Research & Development*, vol. 19, no. 3, pp. 444-448, 2015.

- [9] A. A. El-Habeeb and M. S. Refat, " Synthesis, structure interpretation, antimicrobial and anticancer studies of tranexamic acid complexes towards Ga (III), W(VI), Y(III) and Si (IV) metal ions," *Journal of Molecular Structure*, vol. 2019, no. 1175, pp. 65-72, 2019.
- [10] A. A. S. Al-Hamdani and Z. A. A. Hasan, " Spectroscopic Studies and Thermal Analysis of New Azo Dyes Ligands and their Complexes with some Transition of Metal Ions," *Journal of Baghdad Science*, vol. 13, no. 3, pp. 511-523, 2016.
- [11] A. A. S. Al-Hamdani, A. M. Balkhi, A. Falah and Sh. A. Shaker, " Synthesis and investigation of thermal properties of vanadyl complexes with azo-containing Schiff-base dyes," *Journal of Saudi Chemical Society*, vol. 20, no. 5, pp. 487-501, 2016.
- [12] W. Al Zoubi, A. A. S. Al-Hamdani, S. D. Ahmed and Y. G. Ko, " A new azo-Schiff base: Synthesis, characterization, biological activity and theoretical studies of its complexes," *Applied Organometallic Chemistry*, vol. 32, no. 1, pp. 1-15, 2018.
- [13] A. A. S. Al-Hamdani, " Metal complexes of multidentate Schiff base-azo ligand: synthesis, characterization and biological activity," *Dirasat: Pure Sciences*, vol. 39, no. 1, pp. 61-72, 2011.
- [14] M. Singh and S. Anant, "Synthesis and Spectral Characterization of Praseodymium (III) Complex with New Amino Acid-Based Azo Dye," *International Journal of Inorganic Chemistry*, Vol. 2012, Article ID 767080, pp. 1-6, 2012.
- [15] N. M. Wannas, A. A. S. Al-Hamdani and W. Al-Zoubi, " Spectroscopic characterization for new complexes with 2,2'- (5,5-dimethylcyclohexane-1,3-diyldiene) bis (azan-1-yl- 1-ylidene) dibenzoic acid," *Journal of Physical Organic Chemistry*, vol. 33, no. 11, pp. 1-12, 2020.
- [16] A. B. P. Lever, *Inorganic Electronic Spectroscopy*, Elsevier Publishing Company: Amsterdam, London, 1968, p. 121, edn6.
- [17] A. A. S. Al-Hamdani, A. M. Balkhi, A. Falah and S. A. Shaker, " new azo-schiff base derived with Ni (II), Co (II), Cu (II), Pd (II) and Pt (IV) complexes: preparation, spectroscopic investigation, structural studies and biological activity," *Journal of the Chilean Chemical Society*, vol. 60, no. 1, pp. 1-12, 2015.
- [18] V. T. Suleman, A. A. S. Al-Hamdani, S. D. Ahmed, V. Y. Jirjees, M. E. Khan, A. Dib, W. Al Zoubi and Y. G. Ko, " Phosphorus Schiff base ligand and its complexes: Experimental and theoretical investigations," *Applied Organometallic Chemistry*, vol. 34, no. 4, pp. 1-16, 2020.
- [19] R. M. Silverstein, G. C. Bassler and T. C. Morrill, *Spectroscopic Identification of Organic Compounds*. Wiley: New York, 1981, edn4.
- [20] R. Kh. H. Al-Daffaay , " Preparation, Spectroscopic Characterization of Transition Metal Complexes with Schiff base 2-[1-(1H-indol-3-yl)ethylimino) methyl]naphthalene-1-ol " *Baghdad Science Journal*, vol. 2022, no. 7, pp. 1036- 1044, 2022.
- [21] K. Nakamoto, *Infrared and Raman Spectra of Inorganic and Coordination Compounds*, Wiley-Inter Science: New York. 1997.
- [22] M. J. Kareem, A. A. S. Al-Hamdani, Y. G. Ko, W. Al Zoubi and S. G. Mohammed, " Synthesis, characterization, and determination antioxidant activities for new Schiff base complexes derived from 2-(1H-indol-3-yl)-ethylamine and metal ion complexes," *Journal of Molecular Structure*, vol. 1231, no. 5, pp. 1-30, 2021.
- [23] S. M. H. Obaid, J. S. Sultan and A. A. S. Al-Hamdani, " Synthesis, Characterization and Biological Efficacies from Some New Dinuclear Metal Complexes for Base 3-(3, 4-Dihydroxy-phenyl)-2-[(2-hydroxy-3-methylperoxy-benzylidene)-amino]-2-methyl Propionic Acid," *Indonesian Journal of Chemistry*, vol. 20, no. 6, pp. 1311-1322, 2020.
- [24] A. A. S. Al-Hamdani, N. R. Al-Khafaji and N. Shaalan, " Preparation, Spectral, Thermal and Bio Activity Studies of Azo Dyes Complexes," *Research Journal of Pharmaceutical, Biological and Chemical Sciences*, vol. 8, no. 3, pp. 740-750, 2017.
- [25] A. A. S. Al-Hamdani and W. Al Zoubi, " New metal complexes of N<sub>3</sub> tridentate ligand: Synthesis, spectral studies and biological activity," *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, vol. 137, pp. 75-89, 2015.
- [26] A. A. S. Al-Hamdani and R. G. Hamodha, " Transition Metal Complexes with Tridentate Ligand: Preparation, Spectroscopic Characterization, Thermal Analysis and Structural Studies," *Journal of Baghdad Science*, vol. 13, no. 4, pp. 770-781, 2016.
- [27] W. Al Zoubi, S. G. Mohamed, A. A. S. Al-Hamdani, A. P. Mahendradhany and Y. G. Ko, " Acyclic and cyclic imines and their metal complexes: recent progress in biomaterials and

- corrosion applications," *Royal Society of Chemistry advances*, Vol. 8, no. 41, pp. 23294-23318, 2018.
- [28] W. Al Zoubi, A. A. S. Al-Hamdani, S. D. Ahmed, H. M. Basheer, R. S. Al-Luhaibi, A. Dib and Y. G. Ko, " Synthesis, characterization, and antioxidant activities of imine compounds," *Journal of Physical Organic Chemistry*, vol. 32, e3916, pp. 1-9, 2018.
- [29] W. Al Zoubi, M. J. Kim, A. A. S. Al-Hamdani, Y. G. Kim and Y. G. Ko, " Phosphorus-based Schiff bases and their complexes as nontoxic antioxidants: Structure–activity relationship and mechanism of action," *Applied Organometallic Chemistry*, vol. 33, no. 1, pp. 1-16, 2019.
- [30] V. Y. Jirjees, A. A. S. Al-Hamdani, N. M. Wannas, A. R. Faeqad, A. Dib and W. Al Zoubi, " Spectroscopic characterization for new model from Schiff base and its complexes," *Journal of Physical Organic Chemistry*, vol. 2020, no. 4169, pp. 1-16, 2020.
- [31] M. J. Kareem, A. A. S. Al-Hamdani, V. Y. Jirjees, M. E. Khan, A. W. Allaf and W. Al Zoubi, " Preparation, spectroscopic study of Schiff base derived from dopamine and metal Ni (II), Pd (II), and Pt (IV) complexes, and activity determination as antioxidants," *The Journal of Physical Organic Chemistry*, vol. 34, no. 3, pp. 1-15, 2020.
- [32] W. Al Zoubi, A. A. S. Al-Hamdani and Y. G. Ko, " Schiff bases and their complexes: Recent progress in thermal analysis," *Separation science and technology*, vol. 52, no. 6, pp. 1052-1069, 2017.
- [33] R. Kh. H. Al-Daffaay and A. A. S. Al-Hamdani, " Synthesis, Characterization, and Thermal Analysis of a New Acidicazo Ligand's Metal Complexes " *Baghdad Science Journal*, vol. 19, no. 3, pp. 121-133, 2022.
- [34] A. M. A. Al-Khazraji and R. A. M. Al Hassani, " Synthesis, Characterization and Spectroscopic Study of New Metal Complexes form Heterocyclic Compounds for Photostability Study," *Systematic Reviews in Pharmacy*, vol. 11, no. 5, pp. 535-555, 2020.
- [35] G. D. Reem, H. M. Walaa and G. M. Gehad, " Metal complexes of tetradentateazo-dye ligand derived from 4,4-oxydianiline: Preparation, structural investigation, biological evaluation and MOE studies," *Applied Organometallic Chemistry*, vol. 34, no. 10, pp. 1-20, 2020.
- [36] A. M. N. Khaleel, " Synthesis and Characterization of Trihydro mono and Dihydrobis (indole-3-acetic acid) Borate Ligands and Some of Their Metal Complexes," *Iraqi Journal of Science*, vol. 56, no. 4A, pp. 2762-2772, 2015.
- [37] H.H. Mihsen, S. K. Abass, M. T. Abed –Alhasan, Z. M. Hassan and A. K. Abbas, " Synthesis, Characterization and Antimicrobial Activities of Mixed Ligand Complexes of Fe (II), Co (II), Ni (II) and Cu (II) Ions Derived from Imine of Benzidine and o-phenylenediammine," *Iraqi Journal of Science*, vol. 61, no. 11, pp. 2762-2775, 2020.